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Title: Microstructural Effects of PETN on Detonator Performance

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Microstructural Effects of PETN on Detonator Performance

Connecting powder characteristics to ultimate performance to facilitate *design for qualification*

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2/23/2021

Strategic Motivation

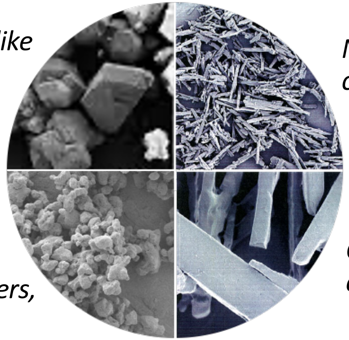
- ~~*Diamond Stamping*~~ *Design for qualification* of HE powder (primarily PETN) for detonator production would reduce production costs and facilitate certification and qualification of detonators.
- ~~Is *diamond stamping* possible?~~ What is required to facilitate *design for qualification?*
- Need to understand:
  - material characteristics important to performance
  - how to most appropriately measure/monitor these characteristics
  - evolution of these characteristics through lifetime and storage conditions

Technical Motivation

- Design, manufacturing, performance, aging, etc. are all dependent upon various material properties, from chemical (species, phase, concentrations, etc.), morphological (particle size, distribution, packing density, etc.) and mechanical (microstructure, moduli, shock propagation, etc.)
- Fisher sub-sieve surface area (FSSA) is the only characterization technique correlating to performance that is routinely in use. *Poorly understood as to why this is the only reliable metric.*
- Loose powder characterization alone is insufficient to understanding performance and aging connections.
- ***Lacking in characterization techniques for consolidated pellets.***
- Currently employed simulation techniques cannot explicitly capture microstructural physics. New FDEM capability can directly utilize microstructure, shedding light on important connections to performance.

Compact block-like crystals

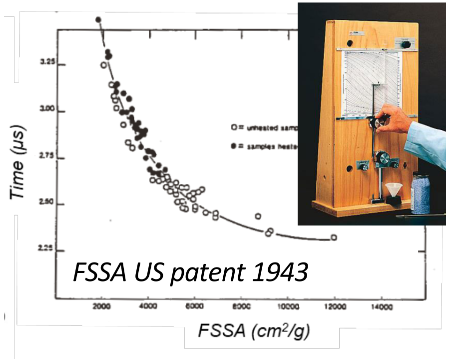
Needle-like crystals



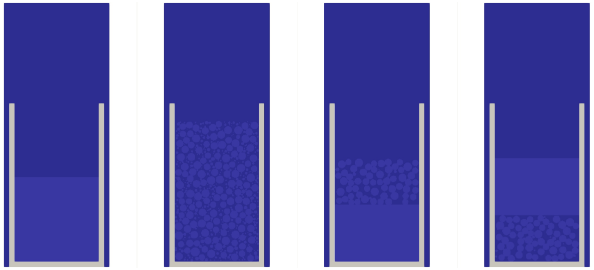
Spray-dried (spheroidal clusters, ~~amorphous?~~)

Crystalline columns and "straws"

PETN particle shapes/sizes create complex microstructures



FSSA correlates to performance but is NOT a microstructural characterization



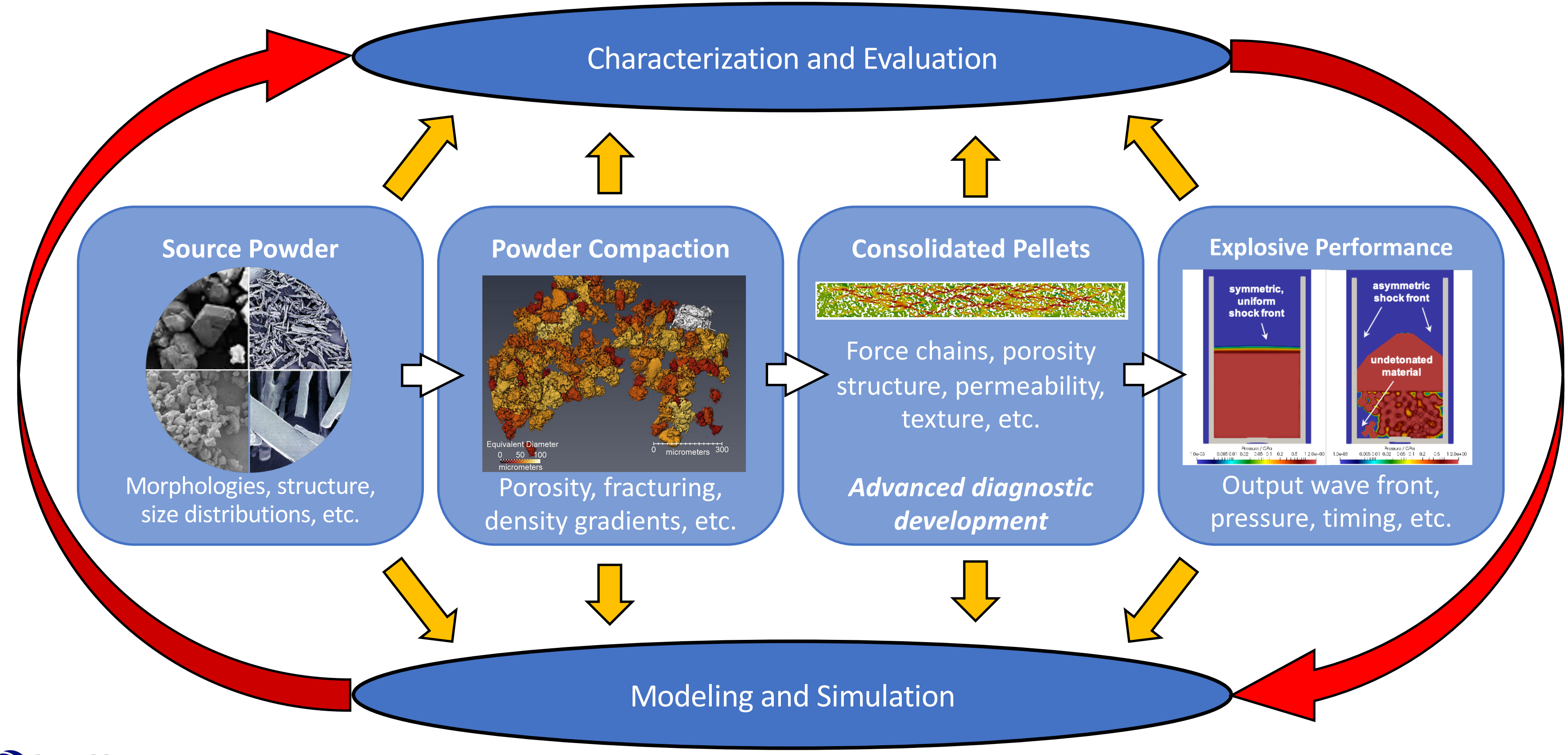
Microstructure (here, porosity and force chains) affect initiation and propagation

Proposal Concept

- Microstructure arises from a combination source material (particle morphology) and processing (e.g., pressing).
- Microstructure affects final performance characteristics

- Goals:
  - Provide new characterization methods for pellet form
  - Understand the effect of powder characteristics on the microstructure and performance of detonator pellets
  - Use new understanding to determine suitable replacement for the Fisher measurement, if possible.
  - Provide predictive modeling capability for future design, certification and qualification efforts. [***complimentary** to full-scale detonator modeling effort, **not** duplicative*]

Integration Concept Overview



Recent Developments for Pellet Characterization

- Resonant Ultrasound Spectropscopy (Material Properties)
- Nonlinear Resonant Ultrasound Spectroscopy (Material Integrity)

Development conducted using Mock HE (900-24, IDOX, and PETCA)

Summary of Report LA-CP-21-20935: *Resonant Ultrasound Spectroscopy on Mock High Explosive Pellets*

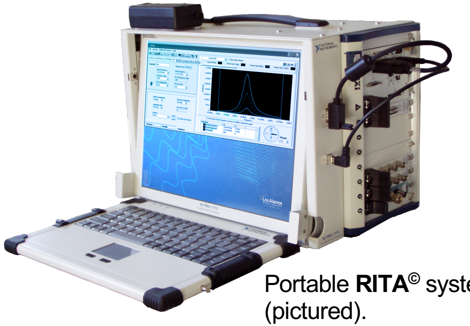
Enabling Technology (Coming to TA-9 summer '21): Resonance Inspection Techniques & Analyses (RITA©)

Using vibrational resonant response of a structure for:

ARS: Acoustic Resonance Spectroscopy for signature identification and "finger-printing"

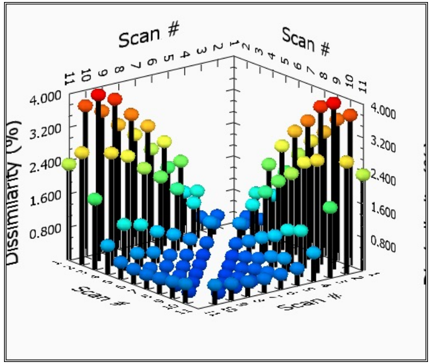
RUS: Resonant Ultrasound Spectroscopy for measuring material properties (elastic constants, density)

NRUS: Nonlinear RUS for damage detection & quantification



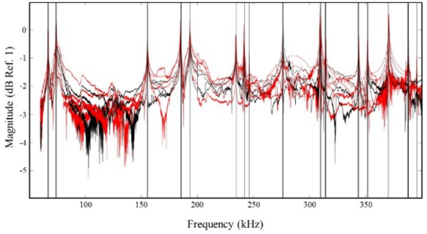
Portable **RITA©** system (pictured).

ARS



Quick sorting of parts

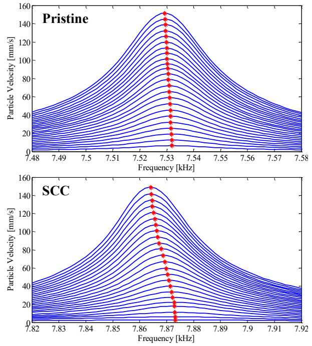
RUS



Material properties (moduli, wave speeds, density)

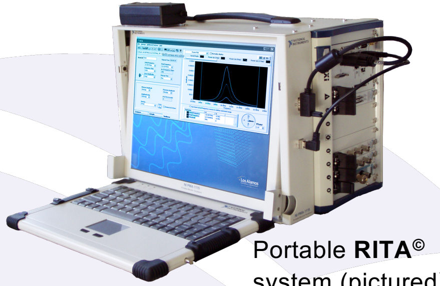
$C_{ijkl}, v_{L,S}, \rho$

NRUS



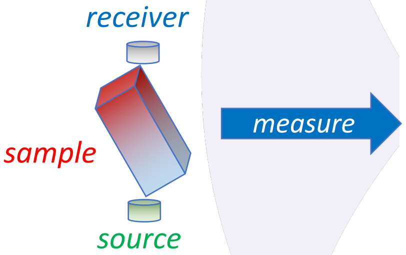
Material Integrity Quantification and Damage Monitoring

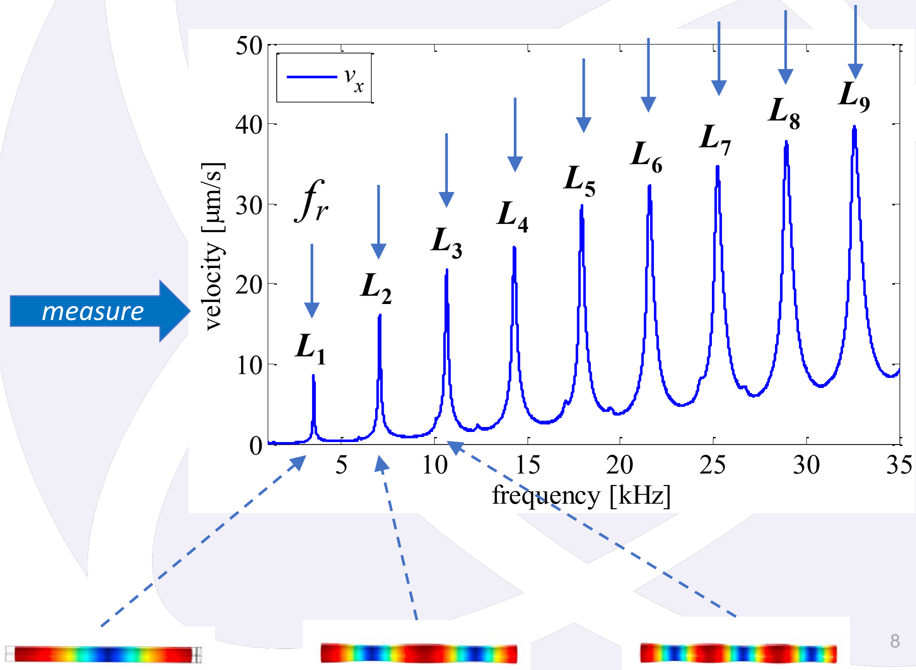
Resonant Ultrasound Spectroscopy (RUS)



Portable **RITA**© system (pictured).

- Measuring resonance requires:
  - Source of vibration (e.g., stepped sine, noise, impulse).
  - Vibration sensor (scanning laser vibrometry strongly preferred)
- Resonance spectra depend upon:
  - material properties (elastic tensor C_{ij}, mass density ρ),
  - Geometry ($\Gamma(x,y,z)$), and
  - boundary conditions (BC).
- Obtaining properties (material, geometric, BC) requires:
  - Vibrational measurements (mode shapes and frequencies)
  - Precise geometry
  - Numerical inversion scheme





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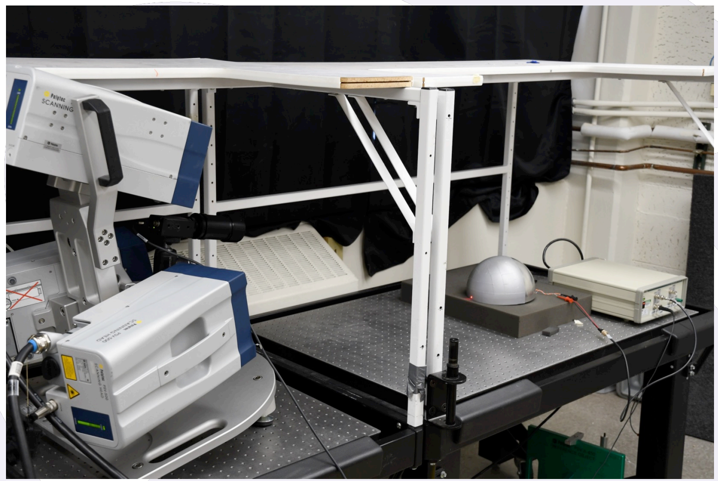
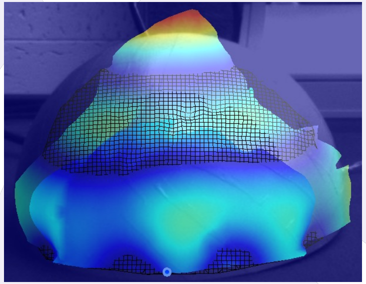
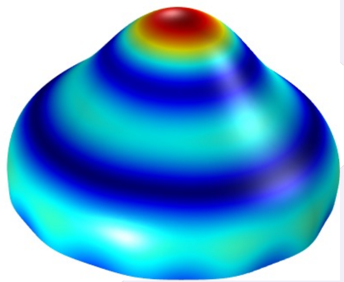
Enhanced RUS (Recent Advancements)

- Scanning Laser Vibrometry: *(measurement)*
  - Enables modal identification
  - Enables non-contact measurements
- Finite Element Modeling: *(computational)*
  - Enables complex geometries and boundary conditions
  - Enables multi-material RUS
  - Required to take advantage of modal ID from laser vibrometry
- Robust Inversion Schemes: *(algorithmic)*
  - Provides greater confidence in results
  - Allows for inversion of density and geometry

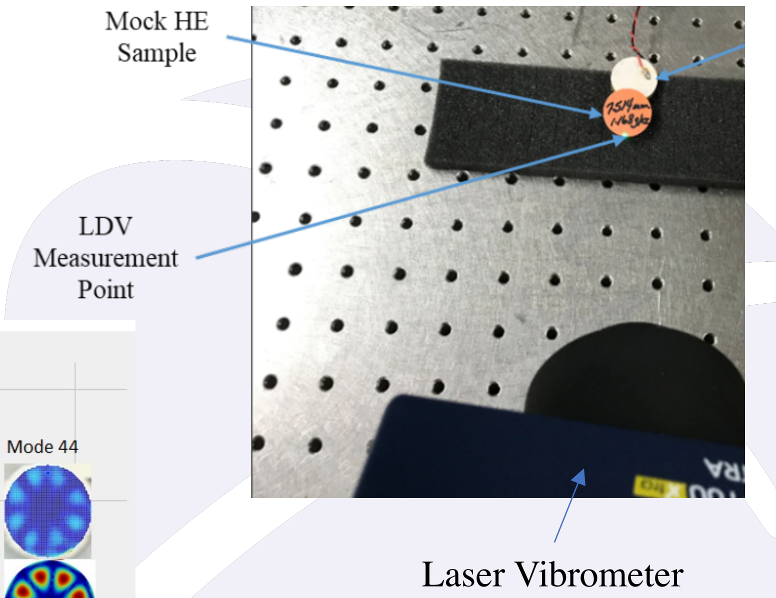


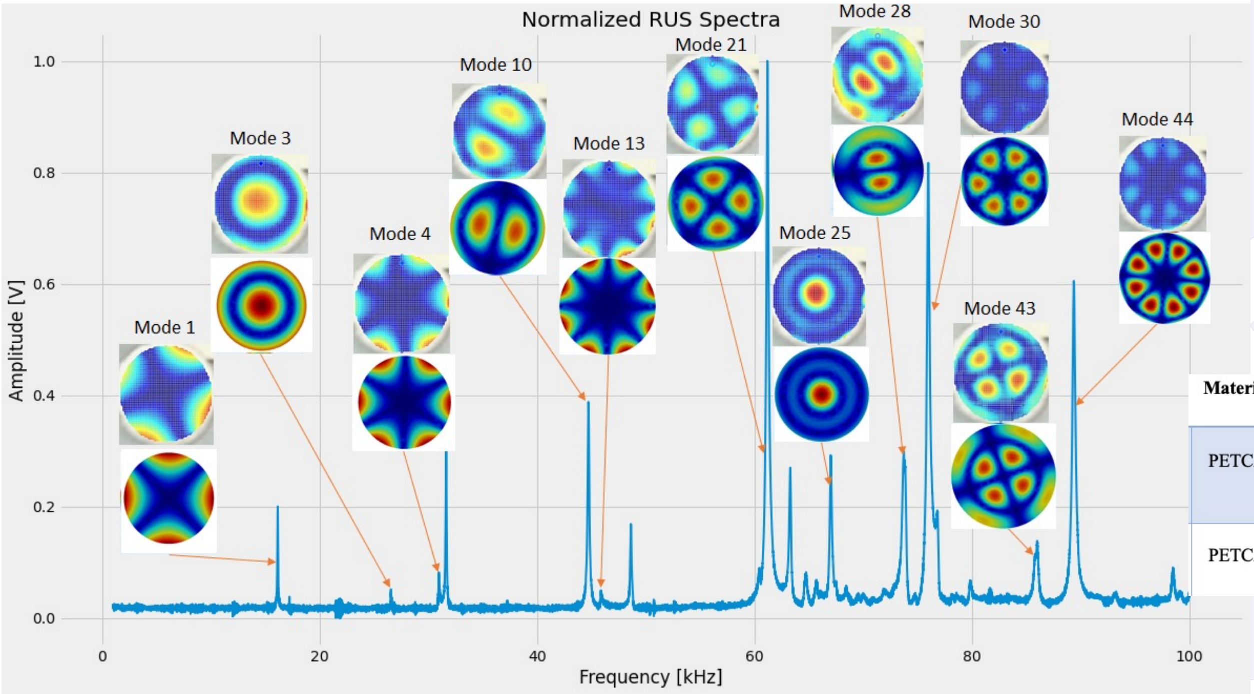




Application of RUS to Mock HE

Example: pellet pressed into an Al cup
Determine: Elastic properties and density

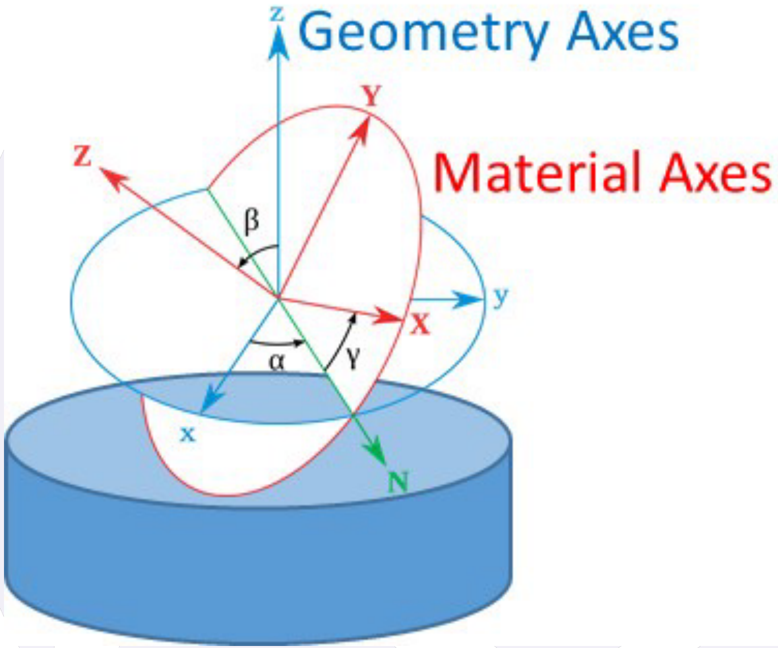




Material	Density (kg/m³)	C_{11} (GPa)	C_{44} (GPa)	Young's modulus (GPa)	Poisson's ratio	RMS Error (%)
PETCA	1176.71	6.21	1.34	3.65	0.362	0.2737
PETCA	1154.0	6.24	1.31	3.59	0.367	0.30181

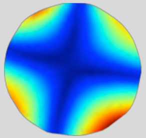
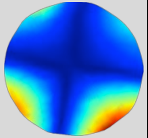
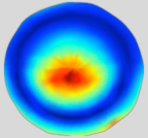
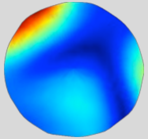
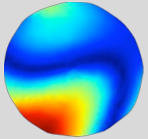
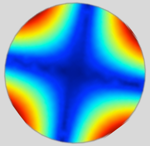
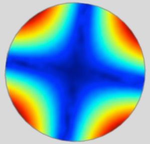
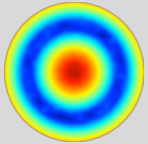
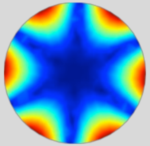
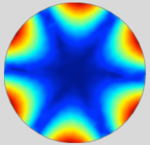
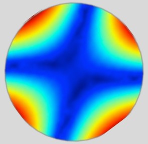
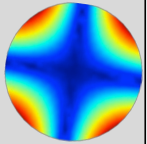
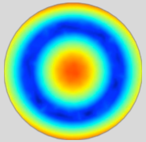
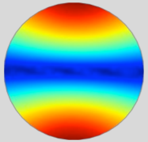
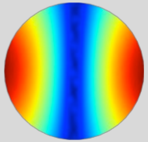
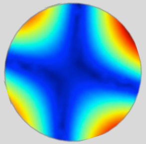
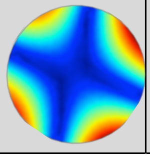
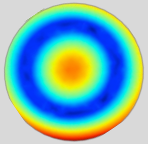
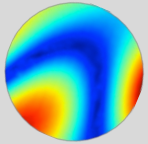
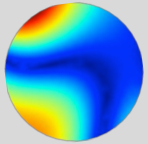
Application of RUS for Anisotropy

- HE pellets are polycrystalline aggregates consolidated through pressing.
- Single crystal properties are anisotropic.
- Random orientation of crystals in pellet *usually* result in isotropic properties.
- However, texturing can occur, e.g., due to off axis pressing, particle morphology, or other material and/or processing variations.



Anisotropy and texture in pressed pellet from RUS

- Anisotropy and texture changes the observed modal vibrations

Data	Material Type	Mode 1	Mode 2	Mode 3	Mode 6	Mode 7	Inversion RMS Error
Measured	NA						NA
Simulated	Isotropic						1.69%
Simulated	Transverse Isotropic						3.25%
Simulated	Transverse Isotropic Euler Rotation						1.57%

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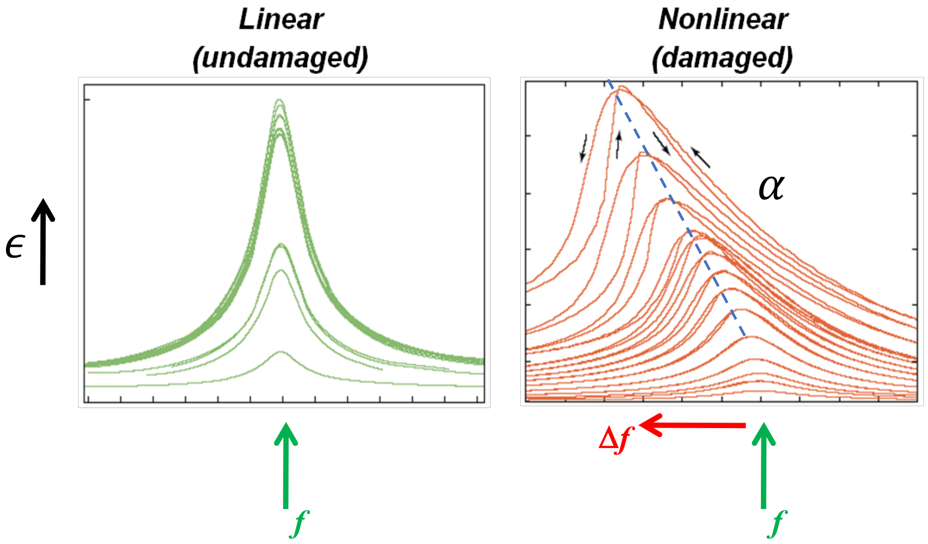
Resulting properties from RUS inversion

- Modal ID is an essential enhancement for determining properties in the presence of anisotropy and texture.

Elastic Symmetry	C_{11} (GPa)	C_{44} (GPa)	C_{33} (GPa)	C_{66} (GPa)	C_{13} (GPa)	α (deg.)	β (deg.)	γ (deg.)	ρ (kg/m³)	Err (%)
Isotropic	2.35	1.03	--	--	--	--	--	--	1837	1.69
Transverse Isotropic	3.11	0.98	8.63	5.82	2.58	0.0	0.0	--	1837	3.25
Rotated Transverse Isotropic	3.23	0.96	8.15	5.81	2.98	53.68	12.56	--	1837	1.57

Nonlinear Resonant Ultrasound Spectroscopy (NRUS)

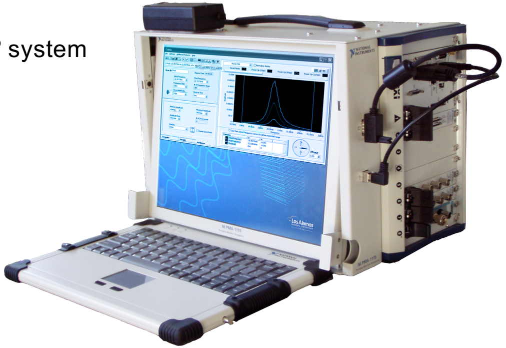
- Quantify hysteretic nonlinear elastic parameter (α) from the natural resonances (f, Δf) of an object driven at multiple strain (ε) amplitudes.



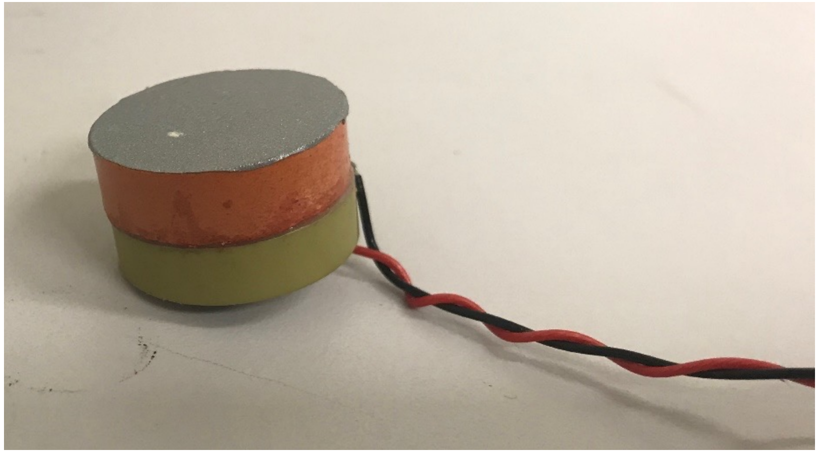
$$\frac{\Delta f}{f} = \alpha\epsilon$$

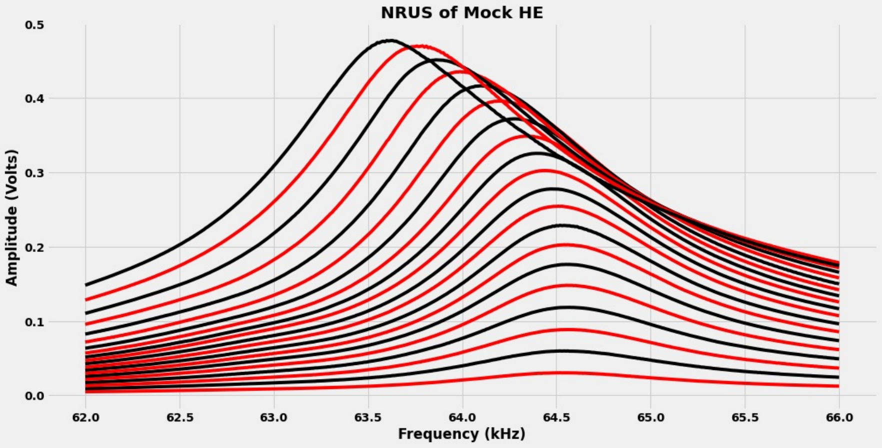
Resonance Inspection Techniques & Analyses (RITA) system previously developed for testing of weapons components.

Portable **RITA**© system (pictured).



Application of NRUS





- Granular materials typically exhibit nonlinear elastic behavior (i.e., amplitude dependent resonance frequency)
- Degree of nonlinear behavior (i.e., amount of frequency shift) relates to the quality/integrity of the consolidation.
- Can this be used as a performance predictor?

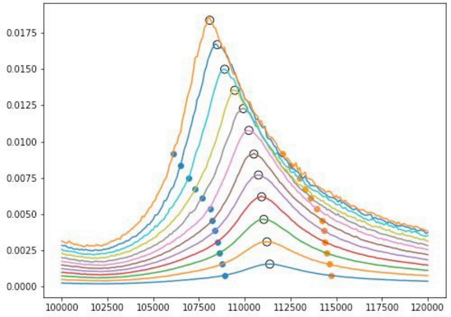
NRUS connection to detonator performance

- NRUS applied to aged PETN pellets

Summary of PEP, Schulze et al (2019)

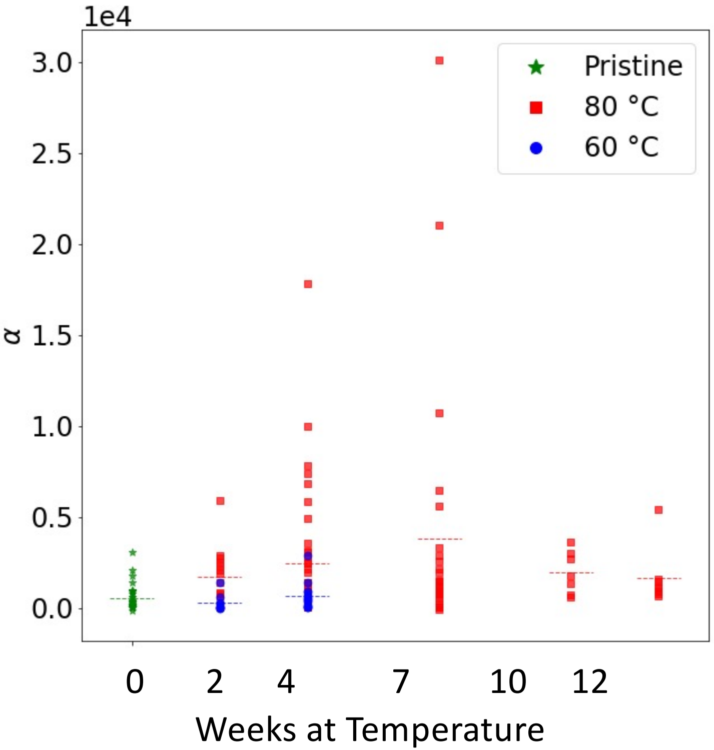
NRUS Example: Thermally Aged PETN pellets

- Can we resolve differences between a pristine pellet and an aged pellet using nonlinear techniques (NRUS)?
- NRUS measurements can be made (below)



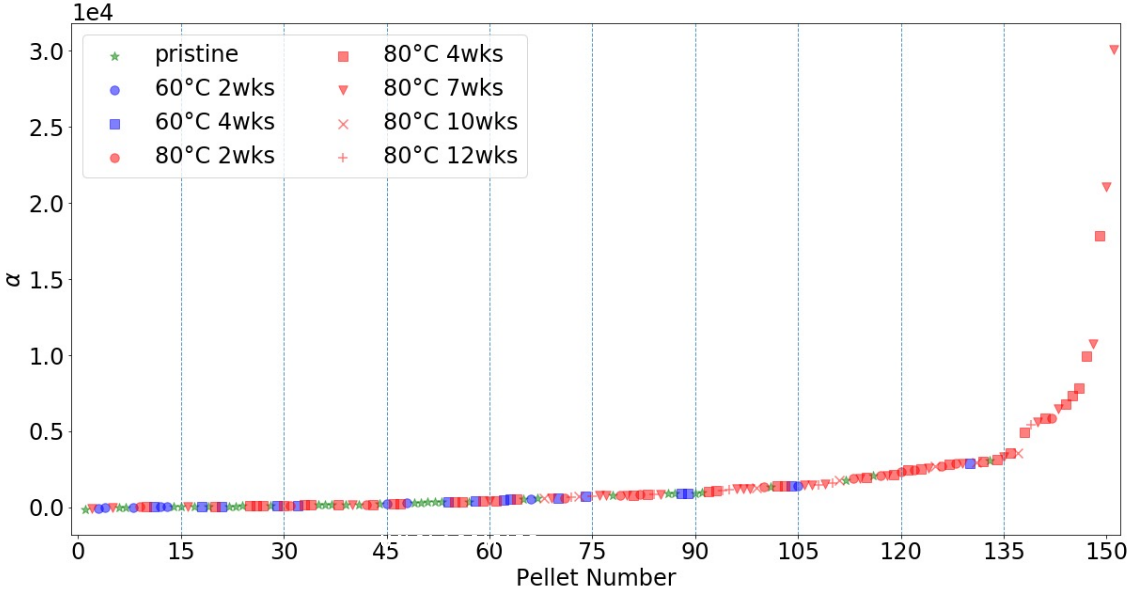
- Results (right) appear to have a lot of variability with aging time, but generally increases in a are observed

Schulze et al (2019)



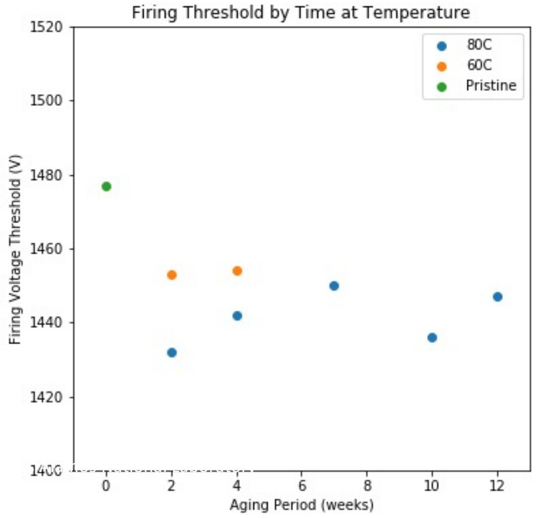
Test Fire Results – Donahue and Schulze

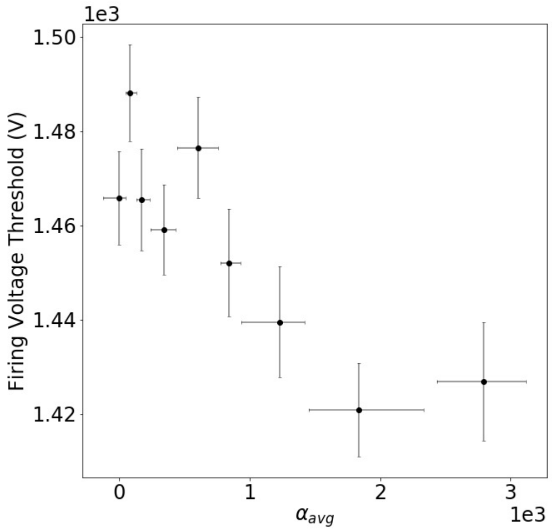
- Rearrange pellets ordered by increasing α.
  - Define ten bins, 15 pellets each,
  - Conduct test fire for threshold determination of each bin.

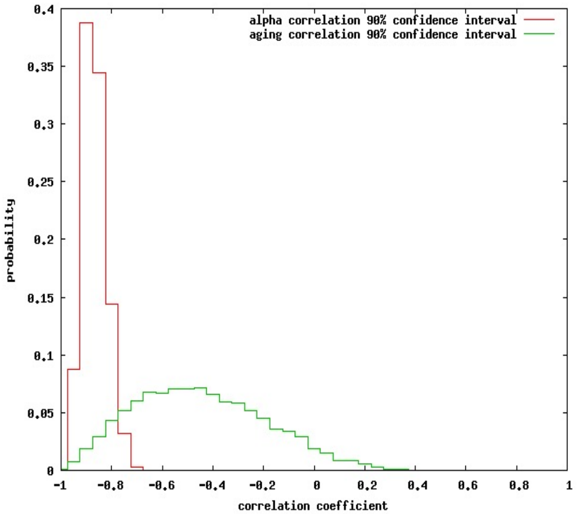


Test Fire Results:

- Firing threshold does not show significant relationship to aging period – left.
- Hysteretic elastic parameter, α, shows strong correlation to performance (threshold) – center.
- Right: correlation between threshold and aging period (green) vs threshold and α (red).







Current Efforts

- Workflow optimizations: (Ph.D. student Luke Beardslee, PSU)
  – Number and location of best measurement locations
  – Automated machine learning (ML) modal ID algorithms
  – Physics informed ML for improved (and faster) inversions
- NRUS inversion for bond/interface properties (e.g., grain-grain contacts) (Post-doc Paul Geimer)
- Connect RUS and NRUS properties to other performance metrics (Ph.D. student Anna Buckthorpe, PSU)
- Application of RUS and NRUS to other detonator materials (e.g., headers, full DCA's) as well as to other systems (e.g., diffusion bonds, on-machine inspection, AM materials/components, vessel qualification, etc.)
- In situ deployment (embedded sensing, micro-reactors, flight testing)